

We claim:

1. A process for distillatively separating mixtures comprising ethylenamines, which comprises carrying out the separation in one or more dividing wall columns.
2. A process as claimed in claim 1, wherein the ethylenamines are ethylenediamine (EDA), piperazine (PIP), diethylenetriamine (DETA), aminoethylethanolamine (AEEA) and/or monoethanolamine (MEOA).
3. A process as claimed in either of claims 1 and 2, wherein the dividing wall column (DWC) in each case has a dividing wall (DW) in the longitudinal direction of the column to form an upper combined column region (1), a lower combined column region (6), a feed section (2, 4) having rectifying section (2) and stripping section (4), and also a withdrawal section (3, 5) having rectifying section (3) and stripping section (5), and the mixture to be separated (feed) is fed in the middle region of the feed section (2, 4), the high boiler fraction is removed via the bottom (bottom draw C), the low boiler fraction is removed via the top (top draw A) and the medium boiler fraction is removed from the middle region of the withdrawal section (3, 5) (side draw B).
4. A process as claimed in any of the preceding claims, wherein the dividing wall column has from 30 to 100 theoretical plates or the dividing wall columns each have from 30 to 100 theoretical plates.
5. A process as claimed in any of the preceding claims, wherein the mixture comprising ethylenamines is a product obtained by reacting MEOA with ammonia and subsequently partly or fully removing ammonia and water.
6. A process as claimed in any of the preceding claims, wherein the mixture comprising ethylenamines is worked up in a dividing wall column in which EDA is obtained as a top product and PIP as a side draw stream at an operating pressure of from 0.1 to 5 bar.
7. A process as claimed in any of the preceding claims, wherein, after the removal of EDA and PIP, further workup is effected in a dividing wall column in which MEOA is obtained as a top product and DETA as a side draw stream at an operating pressure of from 0.01 to 2.5 bar.
8. A process as claimed in any of the preceding claims, wherein, after the removal of EDA, PIP, MEOA and DETA, further workup is effected in a dividing wall

column in which AEEA is obtained as a side draw stream at an operating pressure of from 0.001 to 1.0 bar.

- 5 9. A process as claimed in the preceding claim, wherein the bottom product of the dividing wall column for obtaining AEEA is worked up further in one or more conventional distillation columns to concentrate and purify further higher-boiling ethylenamines and/or ethylenamino alcohols.
- 10 10. A process as claimed in claim 6, wherein the bottom stream of the dividing wall column is worked up further in further conventional distillation columns to obtain first MEOA as a top product in one distillation column and, from the bottom stream of this column, in the next column, DETA is obtained as a top product, and the bottom stream of this column is fed to one or more further conventional columns in order to obtain AEEA, or the bottom stream of this column is fed to a
15 dividing wall column in which AEEA is obtained as a side draw stream.
- 20 11. A process as claimed in any of claims 1 to 5, wherein the mixture comprising ethylenamines is fed to a conventional distillation column in which an EDA/PIP mixture is obtained as a top product, and is separated in a further conventional column into EDA and PIP, and the bottom stream of this column is worked up further in a dividing wall column in such a way that MEOA is obtained as a top product and DETA is obtained as a side draw stream, and the bottom stream of this dividing wall column is fed to one or more conventional distillation columns in order to obtain AEEA, or the bottom stream of this dividing wall column is fed to a
25 further dividing wall column in which AEEA is obtained as a side draw stream.
- 30 12. A process as claimed in any of claims 6 to 10, wherein the upper combined column region (1) of the dividing wall column (DWC) for removing EDA and PIP has from 5 to 50%, the rectifying section (2) of the feed section (2, 4) of the column has from 5 to 50%, the stripping section (4) of the feed section of the column has from 5 to 50%, the rectifying section (3) of the withdrawal section (3, 5) of the column has from 5 to 50%, the stripping section (5) of the withdrawal section of the column has from 5 to 50% and the combined lower region (6) of the column has from 5 to 50%, of the total number of theoretical plates of the column.
35
- 40 13. A process as claimed in any of claims 7 to 9 or 11, wherein the upper combined column region (1) of the dividing wall column (DWC) for removing MEOA and DETA has from 5 to 50%, the rectifying section (2) of the feed section (2, 4) of the column has from 5 to 50%, the stripping section (4) of the feed section of the column has from 5 to 50%, the rectifying section (3) of the withdrawal section (3,

5) of the column has from 5 to 50%, the stripping section (5) of the withdrawal section of the column has from 5 to 50% and the combined lower region (6) of the column has from 5 to 50%, of the total number of theoretical plates of the column.

- 5
14. A process as claimed in any of claims 8 to 11, wherein the upper combined column region (1) of the dividing wall column (DWC) for removing AEEA has from 5 to 50%, the rectifying section (2) of the feed section (2, 4) of the column has from 5 to 50%, the stripping section (4) of the feed section of the column has from 5 to 50%, the rectifying section (3) of the withdrawal section (3, 5) of the column has from 5 to 50%, the stripping section (5) of the withdrawal section of the column has from 5 to 50% and the combined lower region (6) of the column has from 5 to 50%, of the total number of theoretical plates of the column.
- 10
15. A process as claimed in any of claims 2 to 14, wherein the sum of the number of theoretical plates of the subregions (2) and (4) in the feed section in the dividing wall column (DWC) is from 80 to 110% of the sum of the number of plates of the subregions (3) and (5) in the withdrawal section.
- 15
16. A process as claimed in any of claims 6 to 15, wherein the feed point and the side draw point of the dividing wall column for removing EDA and PIP are disposed at a different height in the column with regard to the position of the theoretical plates by the feed point differing from the side draw point by from 1 to 10 theoretical plates.
- 20
17. A process as claimed in any of claims 7 to 16, wherein the feed point and the side draw point of the dividing wall column for removing MEOA and DETA are disposed at a different height in the column with regard to the position of the theoretical plates by the feed point differing from the side draw point by from 1 to 20 theoretical plates.
- 25
18. A process as claimed in any of claims 8 to 17, wherein the feed point and the side draw point of the dividing wall column for removing AEEA are disposed at a different height in the column with regard to the position of the theoretical plates by the feed point differing from the side draw point by from 1 to 20 theoretical plates.
- 30
19. A process as claimed in any of claims 3 to 18, wherein the subregion of the column (DWC) which is divided by the dividing wall (DW) and consists of the subregions 2, 3, 4 and 5 or parts thereof is charged with structured packings or random
- 35
- 40

packings and the dividing wall is designed with heat insulation in these subregions.

- 5 20. A process as claimed in any of claims 3 to 19, wherein the subregion of the column (DWC) which is divided by the dividing wall (DW) and consists of the subregions 2, 3, 4 and 5 or parts thereof is charged with trays and the dividing wall is designed with heat insulation in these subregions.
- 10 21. A process as claimed in any of claims 3 to 20, wherein the medium boiler fraction is withdrawn in liquid form at the side draw point.
22. A process as claimed in any of claims 3 to 20, wherein the medium boiler fraction is withdrawn in gaseous form at the side draw point.
- 15 23. A process as claimed in any of claims 3 to 22, wherein the vapor flow rate at the lower end of the dividing wall (DW) is adjusted by the selection and/or dimensioning of the separating internals and/or the installation of pressure drop-inducing apparatus, for example of perforated plates, in such a way that the ratio of the vapor flow rate in the feed section to that of the withdrawal section is from 0.8 to 20 1.2.
24. A process as claimed in any of claims 3 to 23, wherein the liquid descending out of the upper combined region (1) of the column is collected in a collecting chamber disposed in the column or outside the column and is precisely divided by a 25 fixed setting or control at the upper end of the dividing wall (DW) in such a way that the ratio of the liquid flow rate to the feed section to that to the stripping section is from 0.1 to 1.0.
- 30 25. A process as claimed in any of claims 3 to 24, wherein the liquid is conveyed to the feed section (feed) via a pump or is introduced with flow control using a static feed head of at least 1 m, and the control is adjusted in such a way that the amount of liquid introduced to the feed section cannot fall below 30% of the normal value.
- 35 26. A process as claimed in any of claims 3 to 25, wherein the division of the liquid descending out of the subregion 3 in the withdrawal section of the column to the side draw and to the subregion 5 is adjusted by a control in the withdrawal section of the column in such a way that the amount of liquid introduced to the subregion 5 cannot fall below 30% of the normal value.

27. A process as claimed in any of the preceding claims, wherein the dividing wall column (DWC) has sampling means at the upper and lower end of the dividing wall (DW) and liquid or gaseous samples are taken from the column continuously or at time intervals and investigated with regard to their composition.
- 5
28. A process as claimed in any of the preceding claims, wherein the division ratio of the liquid at the upper end of the dividing wall (DW) is adjusted in such a way that the concentration of those components of the high boiler fraction for which a certain limiting value for the concentration is to be achieved in the side draw, in the liquid at the upper end of the dividing wall, is from 5 to 75% of the value which is to be achieved in the side draw product, and the liquid division is adjusted to the effect that more liquid is passed to the feed section at higher contents of components of the high boiler fraction, and less liquid at lower contents of components of the high boiler fraction.
- 10
29. A process as claimed in any of claims 3 to 28, wherein the heating output in the evaporator is adjusted in such a way that the concentration of those components of the low boiler fraction for which a certain limiting value for the concentration is to be achieved in the side draw, at the lower end of the dividing wall (DW), is adjusted in such a way that the concentration of components of the low boiler fraction in the liquid at the lower end of the dividing wall is from 10 to 99% of the value which is to be achieved in the side draw product, and the heating output is adjusted to the effect that the heating output is increased at a higher content of components of the low boiler fraction and the heating output is reduced at a lower content of components of the low boiler fraction.
- 15
- 20
- 25
30. A process as claimed in any of claims 3 to 29, wherein the distillate is withdrawn under temperature control and the control temperature used is a measurement point in the subregion 1 of the column which is disposed from 2 to 20 theoretical plates below the upper end of the column.
- 30
31. A process as claimed in any of claims 3 to 30, wherein the bottom product is withdrawn under temperature control and the control temperature used is a measurement point in the subregion 6 of the column which is disposed from 2 to 20 theoretical plates above the lower end of the column.
- 35
32. A process as claimed in any of claims 3 to 31, wherein the side product in the side draw is withdrawn under level control and the control part used is the liquid level in the evaporator.

33. A process as claimed in any of the preceding claims, wherein, instead of a dividing wall column, a connection of two distillation columns in the form of a thermal coupling is used.
- 5 34. A process as claimed in the preceding claim, wherein the two thermally coupled distillation columns are each equipped with a dedicated evaporator and condenser.
- 10 35. A process as claimed in either of the two preceding claims, wherein the two thermally coupled columns are operated at different pressures and only liquids are conveyed in the connection streams between the two columns.
- 15 36. A process as claimed in any of the three preceding claims, wherein the bottom stream of the first column is partly or fully evaporated in an additional evaporator and subsequently fed to the second column in biphasic form or in the form of a gaseous and of a liquid stream.
- 20 37. A process as claimed in any of the preceding claims, wherein the feed stream to the column (feed) is partly or fully preevaporated and is fed to the column in biphasic form or in the form of a gaseous and of a liquid stream.
- 25 38. A process as claimed in any of the preceding claims, wherein the dividing wall is not welded into the column, but rather is configured in the form of loosely inserted and adequately sealed subsegments.
- 30 39. A process as claimed in the preceding claim, wherein the loose dividing wall has internal manholes or removable segments which allow access from one side of the dividing wall to the other side within the column.
- 35 40. A process as claimed in any of claims 3 to 39, wherein the liquid distribution in the individual subregions of the column (DWC) may be deliberately adjusted in a nonuniform manner.
41. A process as claimed in the preceding claim, wherein the liquid is introduced to an increased extent in the wall region in the subregions 2 and 5 and the liquid is introduced to a reduced extent in the wall region in the subregions 3 and 4.